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THE ROLE OF CHEMICAL BONDING IN ADHESION

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Strong positive effects have been found when a polybutadiene layer is adhered to a glass substrate by means of interfacial chemical bonds. The bond fracture energy increased by a factor of over 20 when a reactive polysiloxane coating was applied to the glass in comparison with a similar, but non-reactive, polysiloxane coating and the chemically-bonded layer failed cohesively instead of interfacially. Swelling stresses

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were found to discriminate between strongly bonded systems of
this type. After a swelling period in pentane ranging from 10
minutes to over 72 hours, depending upon the proportion of
chemical bonding introduced, the layer detached spontaneously.
This suggests a possible test method for distinguishing between
bonded systems which would normally all fail by cohesive rupture.
Treatment of glass substrates with a monofunctional polybuta-
diene containing a dimethylmethoxysilane endgroup led to no
significant improvement in adhesion. SEM Studies showed that
less than 5 percent of the surface was covered by the mono-
functional material. Thus it appears that the number of reactive
sites per molecule is an important factor governing elastomer
adhesion.

An unexpectedly-high level of adhesion was observed between
simple hydrocarbon elastomers and certain polymeric substrates:
2,6-dimethyl-1,4-polyphenylene oxide, polysulfone, and poly-
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larger than for other polymeric substrates, e.g., polystyrene
and polyethyleneterephthalate. In contrast, the strength of
adhesion of polybutadiene to various inorganic crystalline
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different results were obtained for different substrates. For
example, adhesion to $CaCO_3$, $(NH_4)_2SO_4$, and CaF_2 was some 6 to
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Introduction

It is generally thought that adhesion is improved by forming chemical bonds between the adherends. For example, coupling agents or adhesion promoters with dual functionality are widely employed to join two materials. However, the real nature of the interface and the extent of chemical coupling achieved is generally rather uncertain. Indeed, it has been questioned whether interfacial chemical bonds are formed at all, and whether they are necessary to obtain a strong joint. Even if some bonding is beneficial, it has been suggested that too high a density of interfacial bonds will make the joint brittle and weak. These questions are discussed more fully in recent reviews by Erickson (1), Kaelble (2), and Cassidy and Yager (3). They explain why a fundamental study of the relation between chemical bonding and adhesion is necessary if further progress is to be made in the science and technology of adhesion.

Existing knowledge about the effect of chemical bonding on adhesive strength is deficient in two ways. First, the existence of chemical bonds in most systems has been inferred rather than demonstrated directly and, second, there has been no direct measurement of the number of bonds formed. Progress

towards such information is summarized in this report. Attention
is also drawn to some anomalous results obtained with certain
plastic and crystalline substrates which call for further study.

Studies Using Glass Substrates

A monofunctional polybutadiene has been prepared having
a number average molecular weight of 150,000 and a dimethyl-
methoxysilane endgroup. Evidence for the presence of the
endgroup includes the following: a) a model reaction with
triethylsilanol under conditions previously established for
formation of siloxanes from trimethylmethoxysilane led to
the expected and immediate formation of methanol, b) the
molecular weight doubled under coupling conditions, and, c) this
polybutadiene was found to react with glass surfaces, forming
widely-separated discrete particles somewhat larger in size
than molecular dimensions. The bonded polybutadiene is appar-
ently so incompatible with glass that it coalesces into particles
rather than spreads over the surface.

When the work of detachment was measured for a layer of
polybutadiene (Diene 35 NFA, Firestone Tire and Rubber Company),
crosslinked in contact with the treated glass slide, no dif-
ference was found between the peel strength for the treated

slide and a control sample, within experimental error. All
samples failed interfacially at about the same low peel force,
about 5 N/m. This is probably because the crosslinked poly-
butadiene overlayer was attached to the glass substrate only
via the silanated polybutadiene, i.e., only infrequently, and
thus the strength of adhesion was not significantly increased
in comparison with a control sample.

In contrast, when chemical bonds were formed between a
p-bromomethylphenylpolysiloxane-coated glass slide and a
dicarboxyterminated liquid polybutadiene (CTB, B.F. Goodrich
Company), a continuous layer of rubber was observed by scanning
electron microscopy, rather than discrete rubber particles.
(Standard procedures for coupling amino acids to chloromethylated
supports in solid peptide synthesis were used to form the bond
(4-7)). In addition, as shown in Table I, a marked increase
in the work of detachment W was noted when compared to un-
treated clean slides or slides coated with p-tolylpolysiloxane.
The latter in fact gave lower values of W than the clean slides,
which suggests that the siloxane coating is functioning as a
release agent. The last column in Table I shows that W and

Table I

Effect of p-Bromomethylphenylpolysiloxane and p-Tolylpolysiloxane
Coatings on Work of Adhesion Between Dicarboxyterminated
Polybutadiene and Glass and on Time for Bond Failure in Pentane

% of silane in Coating Solution			
$-Si-\bigcirc-CH_3$	$-Si-\bigcirc-CH_2Br$	$W(J/m^2)$ [a]	Time for Bond Failure in Pentane(hrs)
0	0	34(I)	∼0.08
100	0	20(I)	∼0.08
50	50	450(C)	∼3
0	100	470(C)	> 72

[a] Determined in a 180° peel test with 0.5 cm per min crosshead speed
and 0.1% dicumyl peroxide cure of the polybutadiene. I = inter-
facial failure. C = cohesive failure of the elastomer.

the different degrees of interfacial bonding could also be related to the time for a bond to fail on swelling in pentane. We conclude that in the case of a silanated surface with dicarboxyterminated polybutadiene the compatibility problem is much reduced. It might also be inferred that the number of functional groups per elastomer molecule is an important factor in developing strong adhesion.

Polymeric Substrates

Initial experiments with chlorosulfonated polystyrene substrates also showed a positive effect of chemical bonding on adhesive strength. When chlorosulfonated polystyrene was reacted with amine terminated polybutadiene (ATB, B. F. Goodrich Company) and then a polybutadiene overlayer was applied and crosslinked in situ with 6% lauroyl peroxide, cohesive failure at the rubber-cloth interface occurred at large peel forces. In contrast, untreated polystyrene slides gave a work of adhesion of 85 J/m^2 and showed interfacial failure at the polystyrene-rubber interface. With a sulfur cure the average value was 51 J/m^2 with interfacial failure at the rubber-rubber interface for the chlorosulfonated slides, compared to only 9 J/m^2 with interfacial failure at the polystyrene-rubber

interface for untreated polystyrene. However, compared to glass substrates polystyrene has several serious disadvantages from an experimental standpoint: (i) the low Tg, ~95°C, restricts the choice of peroxide and cure temperature and prevents use of our preferred curing agent, dicumyl peroxide (which reacts at 150°C), (2) nonuniform functionalization was found to occur for many reactions carried out with vaporized reactants, (3) solvents used for application of the functionalized elastomer either dissolved or crazed the polystyrene slides.

Attempts to use substrates with higher values of Tg led to the unexpected discovery of three substrates (adherends) which showed high adhesion to simple elastomers under conditions where weak bonds would be anticipated; namely, low testing rates, moderate temperatures, and with the elastomer layer lightly crosslinked to prevent liquidlike flow (8). The data are presented in Table II along with typical results for other substrates taken from the literature and some new results with a polystyrene substrate.

The adhesion of the elastomers used here to poly-2,6-dimethyl-1,4-phenylene-oxide (PPO), polysulfone, and poly-carbonate is surprisingly high. With these substrates cohesive failure of the elastomer occurred at high peel forces, whereas with other substrates interfacial failure was observed at low

Table II

Effect of Substrate on Adhesion[a]

Substrate	Elastomer[b]	Work of Adhesion (J/m²)[c]		Ref
		50 cpm	25 cpm	
Polyethylene terephthalate[d]	SBR[e]			
	Dicup(1)	-	32(I)	9
	EPR[f]			
	Dicup(1)	-	5(I)	9
Aluminum	SBR[e]			
	Dicup(1)	-	176(I)	9
Pyrex Glass[g]	Polybutadiene[h]			
	Dicup(0.05)	-	609(I)	10
	Dicup(0.2)	-	38(I)	
Quartz	Polybutadiene[h]			
	Dicup(0.05)	-	590(I)	10
Polystyrene[i]	Polybutadiene[h]			
	Lauroyl Peroxide(6.39)	129(I)	-	j
	Sulfur[k]	11(I)	-	
	EPR[l]	120-300(I)	-	
	SBR[m]			
	Lauroyl Peroxide(6)	150(I)	-	
PPO[n]	Polybutadiene[h]			
	Lauroyl Peroxide(10)	~500(c)	-	j
	Dicup(0.1-1)	~460(c)	-	
	Sulfur[k]	76(I)	-	
	EPR[l]	1200-1660(I)	-	
	SBR[m]			
	Lauroyl Peroxide(6)	>1000(c)	-	
Polycarbonate[o]	Polybutadiene[h]			
	Lauroyl Peroxide(6)	~500(c)	-	j
Polysulfone[p]	Polybutadiene[h]			
	Lauroyl Peroxide(6)	~480(c)	-	j
	Dicup(0.1)	~460(c)	-	

Footnotes to Table II

[a] Peel tests at $180°$ were carried out as previously described (8).

[b] The curing agents and the percent of each are listed under the elastomer. Procedures for curing varied and are described for specific elastomer-substrate pairs.

[c] Two speeds are given because literature data are not available at 50 cpm, where our data is most complete. We tested our samples at a variety of crosshead speeds from 0.05 cpm to 50 cpm. When cohesive failure (C) occurred at 50 cpm, it occurred at all speeds used. The differences between 50 cpm and 20 cpm were slight. I = predominantly interfacial failure. cpm = cm per min.

[d] Mylar, Type A (E.I. du Pont de Nemours and Co.).

[e] Ameripol 1513 (Goodrich-Gulf Chemicals, Inc.)-A 60/40: butadiene/styrene cold emulsion SBR rubber. Cured samples were prepared by swelling 1 phr Dicup and 1 phr PBNA from diethyl ether into a thin sheet of rubber, drying, and curing after bond formation for 30 min. at $150°C$. Dicup=dicumyl peroxide. PBNA = phenyl-β-naphthylamine.

[f] Vistalon 404 (Enjay Chemical Co.).

[g] 7740 Glass (Corning Glass Works).

[h] Diene 35 NFA (Firestone Tire and Rubber Co.)-An anionically polymerized polybutadiene rubber. Cured samples were prepared by mixing with variable amounts of peroxide on an open mill and curing after bond formation. With Dicup cures were carried out for 2 hrs. at $150°C$. With lauroyl peroxide 6 hrs. at $85°C$ was used.

[i] Styron 678 (Dow Chemical Co.).

[j] This work.

Footnotes to Table II(contd.)

[k] Cured samples were prepared by mixing on an open mill and curing after bonding for 9 hrs. at 65°C. The recipe contained 100 parts polybutadiene, 12 parts Butyl Eight, 1.5 parts Altax, 1.5 parts sulfur, 3 parts zinc oxide, and 0.5 parts stearic acid. Interfacial failure in these cases may be due to the presence of sulfur at the interface.

[l] EPR 404 (Enjay Chemical Co.)

[m] Ameripol 1502 (Goodrich-Gulf Chemicals, Inc.)-A 76.5/23.5: butadiene/styrene cold emulsion SBR rubber; cured after bond formation for 6 hrs. at 85°C.

[n] (General Electric Co.)

[o] (Cadillac Plastic and Chemical Co.)

[p] (Scientific Polymer Products, Inc.)

peel forces. Even in the two cases where cohesive failure
was not observed, EPR-PPO, the work of adhesion was more
than six times the work of adhesion for a similar EPR-polystyrene
specimen. Such strong adhesion to certain plastics could
clearly be of technological importance.

The reason for this strong adhesion is at present obscure.
Possibilities include good compatibility between the resins
and polybutadiene, chemical bond formation during the free
radical crosslinking reaction of the elastomer layer, and
specific electronic interaction such as π-π orbital overlap
between the unsaturated elastomer layer and the electron-rich
backbones (11). Differences in compatibility as proposed
by Iyengar and Erickson (12) seem unlikely to be responsible
for the observed effects because polystyrene and PPO have
very similar solubility parameters (13-15) and yet behave
very differently in adhesion studies. Further experiments
are planned to investigate the mechanism of this anomalously-
high adhesion.

Crystalline Substrates

Adhesion between polymers and crystalline substances
is not well understood (16) but it is very important technolog-
ically, especially in the preparation of solid propellant fuels

where a crystalline oxidizer is embedded in a rubber matrix (17).
A fundamental investigation of the adhesion of polybutadiene to
model crystalline substrates has therefore been initiated. De-
tachment energy is determined in 90^0 peel tests using an apparatus
consisting of a sensitive force-gauge mounted on an Instron Tester
with the output exhibited on a recording oscilloscope. Freshly
cleaved crystal surfaces were generally used, although a few
experiments were done with polished surfaces.

Considerable variation was found in the strength of the
adhesive bond between polybutadiene and different crystalline
substrates, as shown in Figure 1. The general level of adhesion
was relatively small, however. The crystals examined so far
include cubic KBr, KCl, NaCl and CaF_2, hexagonal $CaCO_3$, and
rhombic $(NH_4)_2SO_4$. The latter is a possible analog of a typical
propellant grain, rhombic NH_4ClO_4, but it is more stable and
thus more convenient for exploratory studies of adhesion between
an elastomer and a crystalline substrate.

The cubic alkali halides (KBr, KCl, NaCl) showed the lowest
levels of adhesion to the polybutadiene elastomer, near zero.
A slight increase was observed as the size of the singly charged
ions and hence the number of electrons surrounding the nucleus

increased, but the differences were rather small. (The ionic radii are 0.95, 1.33, 1.81, 1.95 $\overset{o}{A}$ for Na^+, K^+, Cl^-, Br^-, respectively (18)). CaF_2, which is also cubic but contains a divalent Ca^{2+} cation in addition to the monovalent F^- anion, resulted in 2-3 times the detachment energy of the other cubic crystals examined. $(NH_4)_2SO_4$, which is rhombic and has the divalent anion SO_4^{2-} and two monovalent NH_4^+ cations, showed an even higher detachment energy than CaF_2. Finally, $CaCO_3$, which is hexagonal and contains both divalent cations and anions, showed the highest detachment energy of the crystals tested so far. (The ionic radii are 0.99, 1.33, 1.43 $\overset{o}{A}$ for Ca^{2+}, F^-, NH_4^+, respectively. CO_3^{2-} and SO_4^{2-} are nonspherical ions, and ionic radii have no meaning for them.) From these few crystals it appears that ionic charge and size are more important than crystal structure in determining the detachment energy of polybutadiene from inorganic crystals.

Finally, very limited comparisons between cleaved and "polished" surfaces combined with SEM examination of the surfaces indicate that adhesion to microscopically rough surfaces is significantly increased over adhesion to smooth surfaces. More

careful experiments with higher quality freshly cleaved crystals did not confirm some preliminary observations that drying of the crystal surfaces is essential to secure a measurable degree of adhesion. Drying was beneficial only with student crystals of NaCl and naturally occurring calcite ($CaCO_3$).

In summary, our limited studies with inorganic crystalline substrates indicate that ionic charge, ionic size, and surface roughness of the adherend are important factors determining the detachment energy of a polybutadiene overlayer. Crystallographic structure and electronegativity differences of the anion in the adherend seem to be less important.

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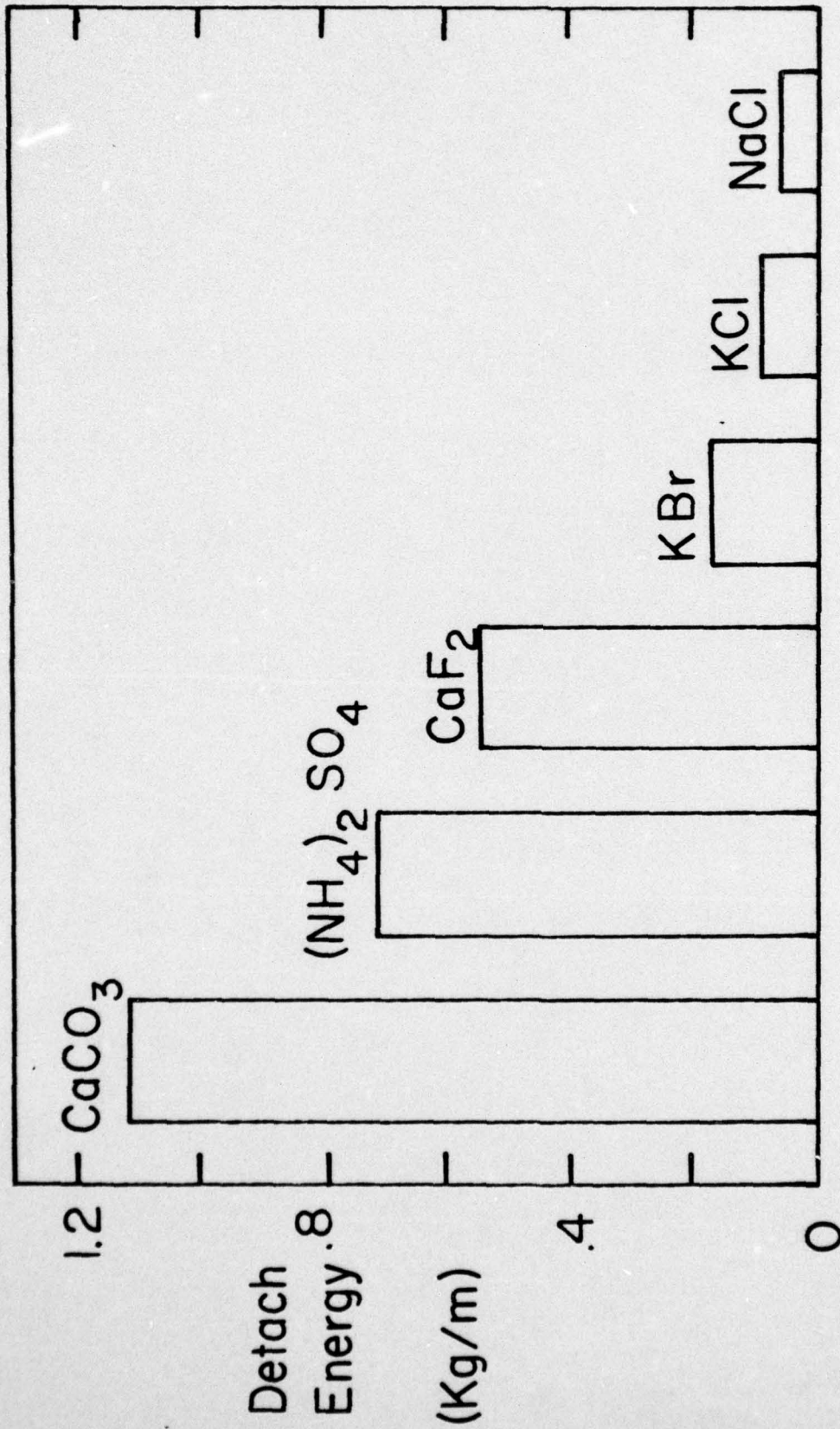
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Figure 1. Variation in strength of adhesive bond between polybutadiene and different crystalline substrates

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